Mine Deactivation Through the Use of a Binding Interlayer

Brian Love
Department of Materials Science and Engineering
Virginia Tech (Mail Code 0237)
Blacksburg, VA 24061
Phone: (540)-231-3186 Fax: (540)-231-8919 E-mail: blove@vt.edu

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LONG-TERM GOAL

The goal of this project was to evaluate how to develop a passive latent lubrication system for mines that would seize over a period of time, allowing for deactivation, even in an armed state. The period of latency was ideally set for approximately two years, although the goal of this project was to see how latency could be established by different mechanisms.

OBJECTIVES

There were three main objectives to address this goal, (1) the use of a microencapsulated resin to cure a lubricating resin system, (2) the use of a viscoelastic resin to perform a mechanical actuation to deactivate the mine, and (3) the third route was an active route using magneto-rheological fluids that are lubricants until exposed to magnetic field.

APPROACH

Epoxy Resins Based on Microencapsulated Curatives. Resins can exhibit a latent increase in viscosity with time if cured by microencapsulated curatives. Isolation of the curing agent by coating it in wax prevents the curing agent from reacting with the base resin until some time, t, when the curative diffuses through the wall of the coating material, or the coating material is dissolved by the use of some solvent in the base resin. Figure 1 shows a schematic representation of an encapsulated curative. Coating materials used for short-term latency generally include lightly cross-linked gelatins and hydrolyzable networks (Welz, 1992, Tabata, 1993). Coating materials for long-term latency generally have a high crystallinity, and include polyethylene waxes and other soluble acrylics (Querat, 1996). Possible curatives for use in this system include any curing agent that reacts by a catalytic mechanism. These include a whole family of imidizole and urea curing agents that are compatible with bisphenol A-based resins. Landec Intelligent Materials is currently the only known commercial source of encapsulated curatives. Landec produces an encapsulated 2-ethyl-4-methyl imidizole curative, as well as a polymer bound imidizole curative. The Landec systems will be examined for their utility in delayed cure applications. The latency of these Landec systems in combination with different resins can be measured by different viscometry methods. Due to the limited source of

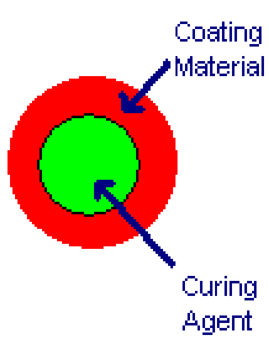

Figure 1: Encapsulated Curing Agent Schematic

commercially available encapsulated curative, there is a need to examine different encapsulation methods. After finding the most effective method, an effort to independently produce encapsulated curatives will begin. (Bank, 1973, Arshady, 1990)

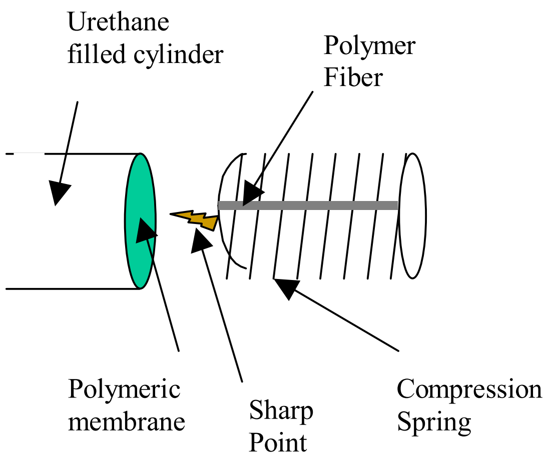

Figure 2: Schematic of the Urethane Foam System

Urethane Based Foam System. Urethane foams can rapidly expand up to 1000% and stiffen when fully cured. The goal here will be to activate pressurized urethane foam at some time, t, by means of a spring-loaded polymeric fiber. Figure 2 shows a representation of the urethane foam system.

The compressed spring loads a polymeric fiber in tension. By design, fiber creep allows the sharp point to puncture a pressurized polymeric membrane. Urethane will foam into the cavity binding the gears in the process. Suitable polymeric fibers for this device include those fibers with a relatively low modulus and low orientation. These fibers include polyethylene, polypropylene, and nylons. Annealing these drawn fibers will increase the strain when compared to an oriented fiber. The candidate urethane foam will be a stable, one-part mixture. These foams generally cure upon reaction with gaseous water vapor. The membrane must prevent foaming inside the cylinder.

Magneto-Rheological (MR) Fluids. MR fluids, composed of magnetic particles suspended in a carrier fluid, thicken in the presence of a magnetic field. The particles (~1um) respond quickly with the applied field. These fluids are used to control the resistance in mechanical systems like fluid filled dampers and brakes. Without a magnetic field the fluid provides little resistance to the motion of a piston or flywheel but as a magnetic field is applied, the viscosity of fluid will increase providing an increasing amount of resistance (Jolly, 1998, Spencer, Carlson).

Properties of MR fluids depend on particle size and concentration and the viscosity of the base fluid. Each formulation will depend on the required yield strength and shelf life of the system. A thicker base fluid results in a longer shelf life, but it may also inhibit the movement of parts while the mine is active. A thinner base fluid with fewer magnetic particles would have less effect on the system while it is active, but may have reduced shelf life due to particle aggregation. Different formulations will be investigated to determine what MR fluid properties are acceptable.

MR fluids can be used as magnetic “switches” binding part movement. Pistons and gears are lubricated without a magnetic field. Once magnetized, the mechanical system becomes more sluggish. One could envision that MR fluids can be used in triggered explosive devices. The device would remain active until a magnet is applied, at which point the fluid would cause moving parts to bind.

WORK COMPLETED

We have worked on three parallel paths to evaluate the viability of each approach as a time delayed increase in mechanical properties. The work on the MR fluid is obviously a more active control system for seizing the lubricant, the other systems have the potential to be passive systems.

RESULTS

Epoxy Resins Based on Encapsulated Curatives. The initial effort examined commercially supplied encapsulated curatives. Due to their rapid curing rate, we compared the cure evolution of the Intelimer curatives mixed with low viscosity resins. Figure 3 shows the cure evolution of the Intelimer 7004 and Intelimer 7024 in Heliox 108 resin (Shell).

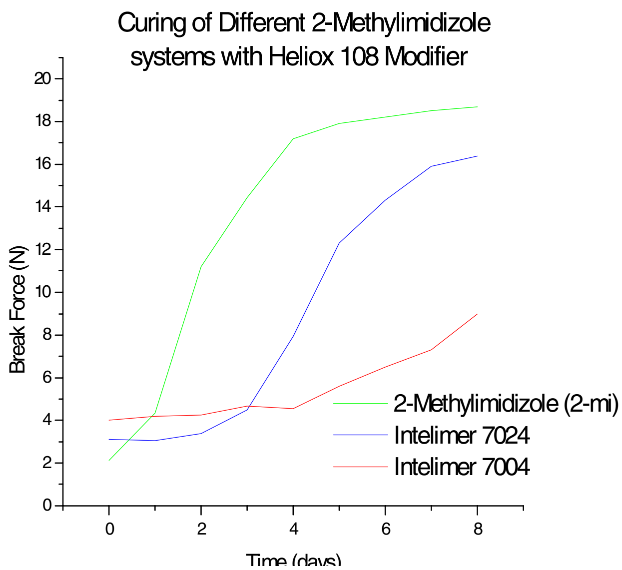

Figure 3: Curing of different curatives in Heliox 108.

The first curative examined, Landec's Intelimer 7024 Encapsulated 2-ethyl-4-methyl imidizole, led to systems with latency of two weeks in a high viscosity resin. The second curative (Intelimer 7004 polymer bound imidizole catalyst) was latent for several weeks in low viscosity resins and for greater than 3 months in high viscosity resins. Table 1 shows the latency of different curatives in several Shell resins. 2-methyl imidizole (2MI) was used to differentiate delayed cure from the standard imidizole curing rate. The viscosity of all resins was measured with a Brookfield viscometer. The Intelimer 7004 curative is labile for more than 5 months. Landec advertises that the stability of Intelimer 7004 in Epon 828 is >0.5-1 year. Further measurements must be taken to assess the ultimate labiality of the 7004 systems. While this curative may meet the labiality requirement of 1-2 years, other encapsulated curatives also need evaluation.

Table 1: Stability of Landec Curatives in Epon Resins

Curing Agent (2%) by weight	**Heliox 107 η = 55-75cP**	**Epon 828 η = 1.1-1.5E3 cP**	**Epon 834 η = 1.45-1.7E3 cP**	**Epon 836 η = 1.6-1.85E3 cP**
Intelimer 7024	4-7 days	2 weeks	2-3 weeks	2-3 weeks
Intelimer 7004	10-14 days	> 5 months	> 3 months	> 3 months
2MI	1-3 days	3-7 days	3-7 days	3-7 days

The next step was to devise a way to manufacture an independent source of curative. Building on the work of Hoffman et al. (Hoffman 1996, US Patent #5,601,761), a melt spinning apparatus was constructed, shown in Figure 4.

A slurry of the curative and coating material is dispensed onto a heated disc rotating at ~6K rpm. The particles are collected and washed in ethanol. Encapsulating efficiency is found by thermogravametric analysis (TGA). The variables for coating curatives include the slurry temperature, the slurry deposition rate, the wheel speed and temperature, and the type of curative and coating material used. We will use polyethylene waxes since their high crystallinity inhibits diffusion through the shell.

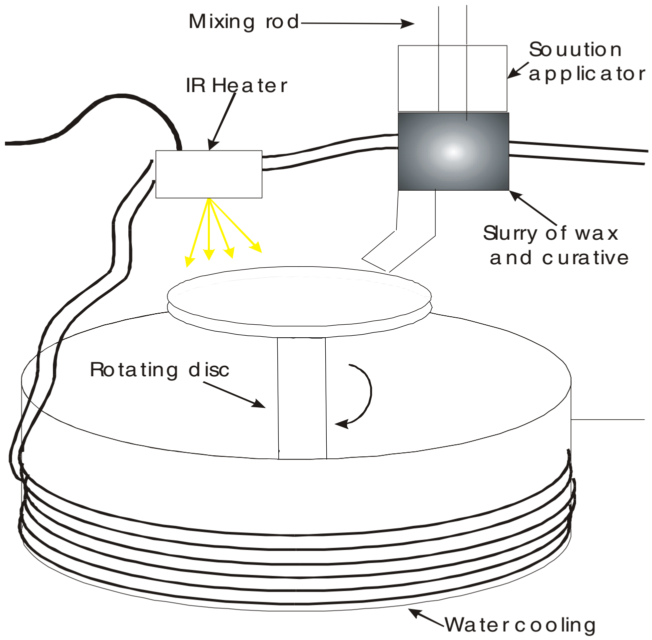

Figure 4: Melt Spin Coating Apparatus

Preliminary experiments have been performed using a blend of Polywax 2000 and 1000, and 2-methyl imidizole was encapsulated at an efficiency of 3-8%. The particle size of the microcapsules ranged from 50-500 µm.

Urethane Based Foam System. Fiber grips were constructed for use in a Stable Microsystems Texture Analyzer cantilever tensile apparatus. The grips prevented the stress concentration and distributed the applied load over the circumference of a round cylinder. The fiber grips were first tested using a single filament Nylon and since that, we have evaluated two types of polypropylene multifilament yarns with different deniers and several other nylon fibers both as received and annealed. Tensile tests were first performed on each of the fibers to determine the tensile strength of each material and from that, an acceptable creep force was determined. Short term Creep tests were then performed on both fibers using a creep force of 5N.

Empirical equations were fit to the experimental creep data similar to the example below for low denier polypropylene. The empirical model fits well with the data set. And from that, extrapolations out to some time t in order to find the total creep distance.

Although this model fits very well with the existing data, it is still uncertain if the curve has the same shape after extended periods of time. Further analysis will need to be done in order to determine long term creep. Advanced models can account for occurrences such as strain hardening and sometimes even polymer degradation. The advanced analysis must be compared with simple curve fitting in order to evaluate the validity of the existing model.

The creep of the nylon fibers was more reproducible than that of the multifilament polypropylene fibers. Annealing caused more fiber creep. Table 2 shows creep's effect by annealing. In this case, annealing the fiber for 90 minutes causes a 70% increase in the creep.

Figure 5: Fiber Grip Assembly

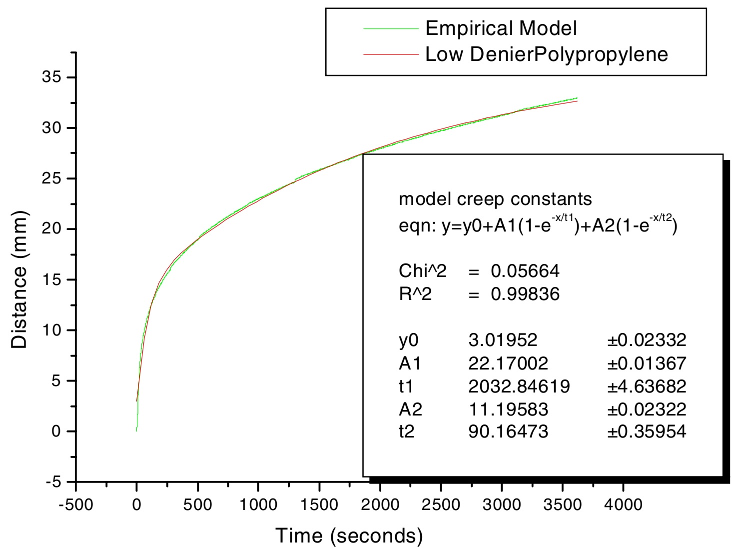

Figure 6: Creep curves for low denier polypropylene

Table 2: Effect of Annealing on Creep for Nylon Fibers

Time annealed at 100C (min.)	Creep over 30 minutes (%)
0	2.8
30	3.3
60	4.1
90	4.8

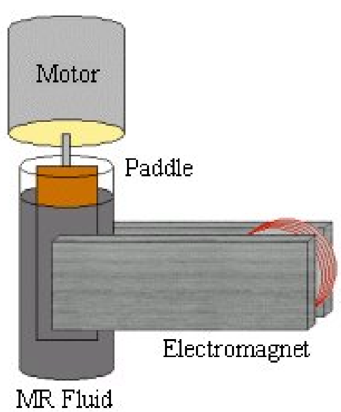

Figure 7: Paddle Submerged in MR Fluid.

MR Fluid. The first attempt at utilizing the MR Fluid was to submerge a rotating paddle and see if the fluid would supply enough torque to stop it. A small 3-volt, Radio Shack hobby motor was used to turn the paddle and an electromagnet surrounded the vial of fluid. Both the speed of the motor and the strength of the magnet can be varied by changing the voltage supplied to each.

This system produced less than desirable results due to the quantity of fluid used and led to the investigation of other ways to contain the fluid.

The next generation MR Fluid scheme used Lord Corporation's MR Sponge technology. The sponge retains fluid where it is needed and prevents the ferrous particles from aggregating. When a magnetic field permeates the sponge, any motion across the sponge is hindered. The MR fluid acts as a lubricant when in the off state. The sponge is being tested in both linear and radial motion as well as with both fixed and electro-magnets.

In Figure 8 below a steel plate is sandwiched between two MR sponges in which a magnetic field is applied. When the magnetic field is off the steel plate moves freely. When the field is on the force required to move the plate increases significantly.

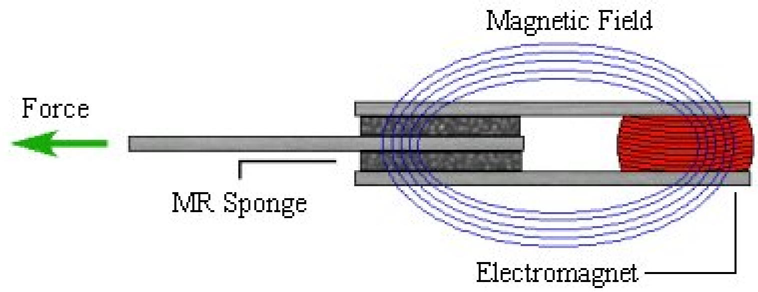

Figure 8: Steel Plate Between MR Sponges.

The next step was to maximize the sponge contact area and minimizing the amount of fluid that needs to be energized. We also hope to energize the fluid more effectively with magnetic fields. The previous setup used a lot of fluid that required a sizable current to saturate the fluid.

To maximize surface area and decrease the overall size of the system the configuration changed from plates sliding across each other to concentric shafts that fit inside one another. The electromagnet will then be wrapped around the outer shaft. This setup will allow the control of rotational motion as well as longitudinal motion. A picture of the setup can be seen in Figure 9.

The open celled foam that was previously used was replaced with a thin loosely woven fabric. The fabric will hold much less fluid and will require less magnetic field to energize.

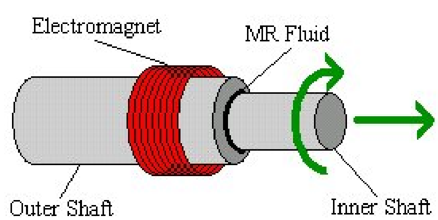

Figure 9: MR Fluid Sponge Between Two Concentric Shafts.

Using a flowing fluid in a glass vial we have generated preliminary values of over 20 gm-cm of torque. By using larger quantities of MR fluid we require greater magnetic fields and more current. This particular setup required a large electro magnet and a 12 Volt automotive battery as the power source. The goal for MR fluid is to now obtain the most force by using the least magnetic field. Minimizing the amount of fluid to energize will produce better results with smaller power requirements.

The results of the MR sponge test are promising as a significant force can be produced without using much fluid. We hope to improve the current design of the sponge to increase efficiency.

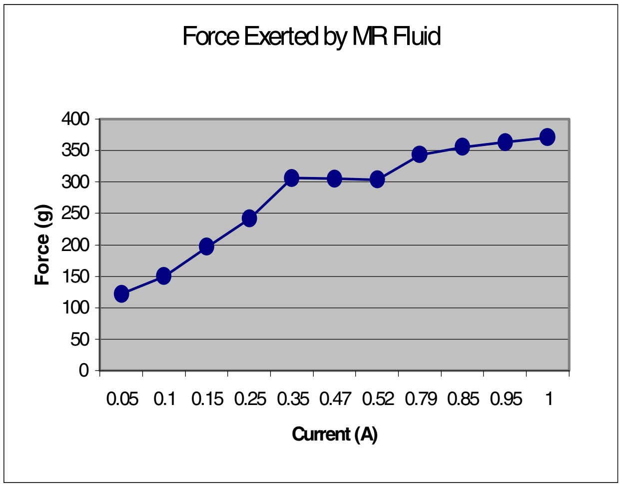

Figure 10: Graph of Force Supplied By MR Sponge

The force exerted by the MR sponge increases directly with current. Here, above 0.4 Amps the force generated leveled off. This is due to the saturation of either the electro magnet or the fluid itself.

Standard 9-volt batteries power the electro magnets for the MR sponge. The magnet dissipates the energy from the batteries too quickly. Hopefully as the system and magnet sizes are scaled down, the required power will less.

The first attempts with the concentric shaft setup did not work properly due to the orientations of the magnetic fields. However the use of the new open-weave fabric shows promise of being scaled down to fit into small devices.

IMPACT/APPLICATIONS

Good progress has been made to evaluate the three different approaches. It appears that the development of a system that can either be passively or actively seized by the use of a thickening lubricant has good potential. The use of the MR fluid may also have tremendous overlap for other actuators and dampers of interest to ONR and we are progressing in establishing the limits of this type of system in terms of size and weight.

TRANSITIONS

With an understanding of how long any of these systems are latent, the torque reduction associated with each system over time should be determinant. This information should allow designers to either use a passively latent system using smaller motors or to bypass this latent fault system by designing with a larger motor. We should also be able to provide input on how much size and mass is incorporated in each fault scenario and this should help in identifying the size and scale of future redesigns of devices.

RELATED PROJECTS

Professor Sean Corcoran at Virginia Tech is also looking at fault systems that use corrosion as the main mechanism of diffusing an actively armed device like a mine. It is possible that a combination approach could also be envisioned.

REFERENCES

M.M. Welz, C.M. Ofner III, 1992. Examination of Self-Crosslinked Gelatin as a Hydrogel for Controlled Release *J. Pharm. Sci*, 81, 85-90.

Y. Tabata, R. Langer, 1993. Polyanhydride Microspheres that Display Near-Constant Release of Water-Soluble Model Drug Compounds, *Pharmaceutical Research*, 10, 391-399.

E. Querat, L. Tighzert, J.-P. Pascault, 1996. Microencapsulation of Isocyanates. Characterization and Storage Stability of Microcapsules in a Polyester α,ϖ-ol, *J. Coatings Tech*., 68, 83-90.

M. Bank, R. Bayless, R. Botham, P. Shank, 1973. Encapsulated Curing Agents for Epoxy Resins, Modern Plastics, November, 84-86.

R. Arshady, 1990. Microspheres and Microcapsules, a Survey of Manufacturing Techniques: Part III: Solvent evaporation, *Poly. Eng. Sci*., 30, 915-924.

Mark R. Jolly, Jonathan W. Bender, J. David Carlson, 1998. Properties and Applications of Commercial Magnetorheological Fluids, SPIE 5th Annual Int. Symposium on Smart Structures and Materials, San Diego, CA.

B.F. Spencer, Jr., S.J. Dyke, M.K. Sain, J.D. Carlson, Phenomenological Model of a Magnetorheological Damper, *ASCE Journal of Engineering Mechanics.*

J.D. Carlson, D.M. Catanzarite, and K.A. St. Clair, Commercial Magnetorheological Fluid Devices, Lord Corporation, Cary, NC.

D.K. Hoffman, D.V. Dellar, 1996. Storage Stable Epoxy Resin Adhesives Based on Encapsulated Curatives, 28th International SAMPE Technical Conference, November 4-7. (Data obtained from Shell Chemical Company)

PATENTS

Hoffman, D.K., et al. U.S. Patent # 5,601,761; Encapsulated Active Materials and Method for Preparing Same, February 11, 1997. (Data obtained from Shell Chemical Company)